Filament-Winding Plastics

Part 2 - Role of the Resin in Glass-Fiber-Reinforced Structures Under Tensile Stress

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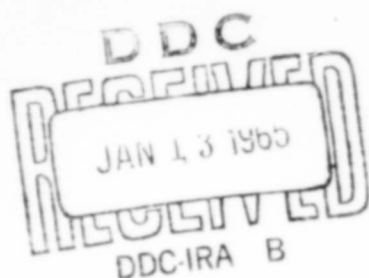*Organic and Biological Chemistry Branch*
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PREVIOUS REPORT IN THIS SERIES

"Filament-Winding Plastics, Part 1 – Molecular Structures
and Tensile Properties," J.R. Griffith and F.S. Whisenhunt,
NRL Report 6047, Mar. 16, 1964.

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ABSTRACT

A study has been conducted on amine-cured epoxy resins and the effects on the ultimate burst strength of internally loaded glass-reinforced filament-wound vessels when these resins were used as the plastic matrices. The tensile properties of the matrix were changed by systematically altering the molecular structure of the amine-cured epoxy resin. It was found that resin tensile properties had an insignificant effect on the ultimate burst strength of a well-designed and well-fabricated vessel. However, it was also found that a matrix of good tensile properties improved the reliability of the burst pressure by providing a healing effect or minor winding errors. The glass stresses at burst of the test vessels were in the 400,000-psi range, which is about the ultimate tensile strength of bundles of "E" glass.

A summary has been prepared which discusses the state of the art and unsolved problems relevant to the plastic matrix in glass-reinforced filament-wound vessels. In particular, the deleterious effect of voids on externally loaded structures and the necessity of removing these voids is discussed.

PROBLEM STATUS

This is an interim report; work is continuing on the problem.

AUTHORIZATION

NRL Problem R05-24C
Project SPO WW-041

FILAMENT-WINDING PLASTICS

PART 2 – ROLE OF THE RESIN IN GLASS-FIBER-REINFORCED STRUCTURES UNDER TENSILE STRESS

INTRODUCTION

With the coming of glass-filament-reinforced plastic rocket-motor cases, there has been much speculation as to the role of the resin in these structures. All such speculations were based on little or no data; a decision was therefore made by the U.S. Naval Research Laboratory to undertake a series of experiments to determine what the resin could contribute to the ultimate burst strength of a filament-wound motor case.

The approach to these experiments was to make an orderly change in the chemical structure of the curing agent of the plastic matrix, in order to create different mechanical properties. This approach was chosen for three reasons.

1. Chemically changing the resin would allow an analysis of the effect of the change in greater detail and depth than would be allowed by the use of additives such as flexibilizers, which are of varying composition and which work through various mechanisms.

2. The curing agent, being a more simple molecule than the basic resin monomer, would be less difficult to alter chemically and could produce sufficient changes in the tensile properties of the cured system.

3. There was a possibility of discovering how tensile properties of a cured system were affected by the placement of chemical groups in the curing agent.

The relation that exists between the tensile properties of the plastic matrix and the ultimate burst strength of a filament-wound vessel was the chief subject of the study. Part 1 of this study (NRL Report 6047, Ref. 1) disclosed some information showing the relation of resin tensile properties to the placement of chemical groups in the curing agent. Similar studies of molecular structure versus resin properties will be the subject of a subsequent report.
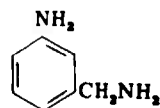
MOLECULAR STRUCTURE

The three curing agents used here, which have been described in detail previously (1), are shown below diagrammatically.
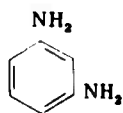
m-Phenylene diamine

(MPDA)
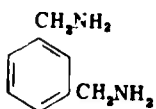
m-Xylylene diamine

(MXDA)

m-Aminobenzylamine

(MABA)

1

Table 1
Tensile Properties of Amine-Cured Diglycidylether of Bisphenol-A Epoxide Plastics

Sample	m-Phenylene diamine			m-Xylylene diamine			m-Aminobenzylamine		
	Tensile (psi)	Elongation (percent)	Elastic Modulus (psi)	Tensile (psi)	Elongation (percent)	Elastic Modulus (psi)	Tensile (psi)	Elongation (percent)	Elastic Modulus (psi)
1	12,900	7.5	427,000	10,800	7.4	410,000	12,300	11.0	426,000
2	12,600	6.8	451,000	10,800	7.8	427,000	12,200	10.3	403,000
3	12,400	6.3	488,000	10,000	4.9	433,000	12,200	10.0	402,000
4	13,000	8.7	451,000	10,800	7.6	445,000	12,100	11.5	410,000
5	12,900	8.1	496,000	10,400	5.6	464,000	12,100	9.5	406,000
Average	12,800	7.5	463,000	10,600	6.7	436,000	12,200	10.5	409,000
Standard Deviation	200		28,000	360		20,400	100		9,700

The compound m-Phenylene diamine is a widely used amine curing agent for epoxy resins. Since MPDA produces a resin of excellent properties, and is more or less a standard curing agent, alterations were made to this structure to produce resins of different tensile properties which would provide the most orderly and easily obtainable comparison of properties versus structure. Curing agents MPDA and MXDA were obtained from commercial sources, while MABA was synthesized at NRL in both laboratory glassware and in pilot-plant equipment. Table 1 shows a comparison of the properties obtained with these three curing agents when used with the diglycidylether of bisphenol-A. The diglycidylether of bisphenol-A is used extensively in filament winding, and was used exclusively as the epoxy monomer in this study. This material, shown below diagrammatically, was obtained commercially in a very pure form.
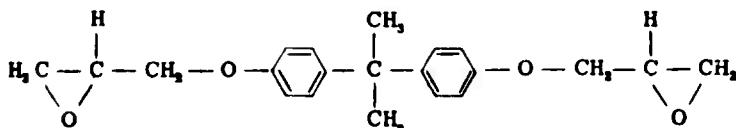
The entire series of experiments was run with one lot of the diglycidylether of bisphenol-A to insure that the small differences in the material which occur from lot to lot would not be a factor in determining the results.

EXPERIMENTAL PROCEDURE

General

The purpose of this study was to determine the effects of the tensile properties of the resin in filament-wound, internally pressurized vessels that were similar in design to rocket-motor cases. This objective required that the test specimen must be loaded biaxially rather than uniaxially, as is the case in a ring specimen. The most practical design for such a test specimen is a closed cylinder that can be given an internal hydraulic load. The design of the test vessel is used with the permission of Lamtex, Inc. All test vessels, which are referred to as "bottles," were wound at NRL on a commercially manufactured winding machine. All the glass used in this study was "E" glass, Owens-Corning G 150 1/O 1z HTS (0.00036 in./average diameter), and was from one lot. At NRL, this glass was stored at 73°F and 40 percent relative humidity, but its predelivery history of storage is not known. All mandrels used in winding the bottles were made at NRL. All
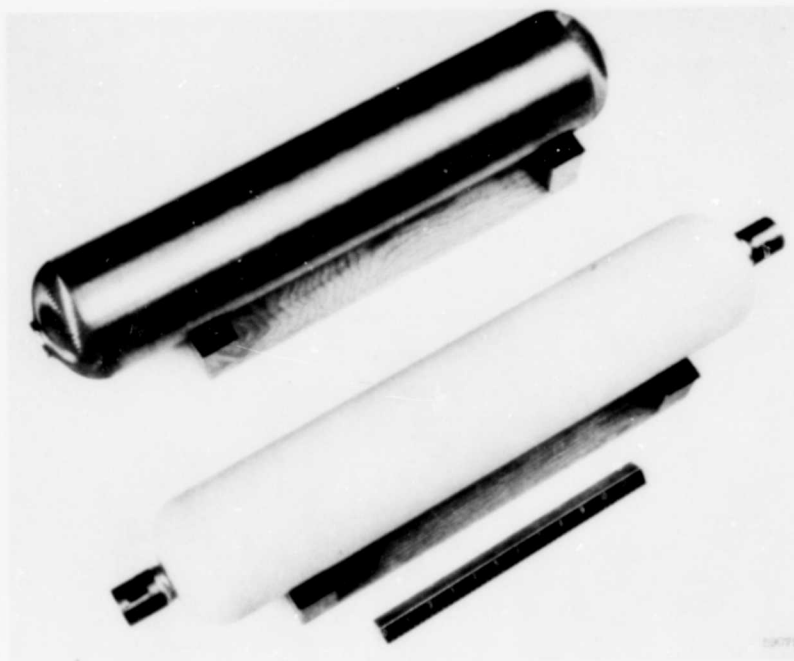
Fig. 1 - A mandrel (below), and finished,
cured bottle (above)

All burst tests were conducted on a servo-controlled hydro-test facility. The details of the bottle manufacture and testing are described in subsequent sections.

Bottle Design

The Lamtex bottle, approximately 25 in. long and 5 in. in diameter, is designed to be an isotensoid, "balanced" structure. The entire test bottle was wound with 30 ends of glass, which makes a band width of 0.225 in., and with a helical winding angle of 25 degrees. The design of the bottle was altered for this study by leaving off one layer of vertical wraps in order to force the failures in the cylindrical section. The wall thickness of the cured bottles was about 0.040 in.

Mandrels

The mandrels (Fig. 1) were cast of Paraplast* No. 33, which is a mixture of water-soluble salts and bases, heavily laden with an inert filler material. Paraplast No. 33 has a melting range of 315° to 330°F, a coefficient of thermal expansion of about 5×10^{-5} in./in./°F, and is dimensionally stable as long as it is stored in an atmosphere of very low relative humidity. To assure uniform dimensions, the mandrels were used soon after manufacture.

When suitable molds and process parameters were obtained, mandrels were produced with a ±0.005-in. tolerance as they came from the mold. This eliminated the need for grinding the mandrels and allowed their production at a relatively low cost ($10 for material and 1/2 man-hour). Liners were developed for prewinding application to the mandrels,
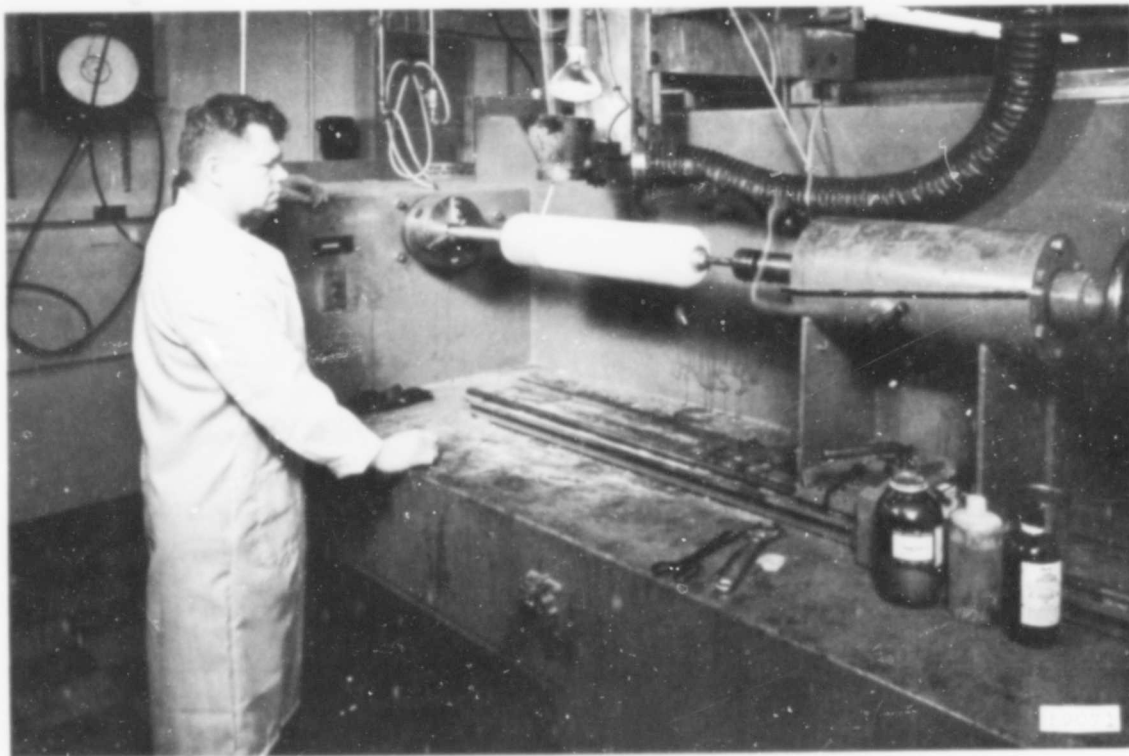
*Trade name of Rezolin, Inc.

Fig. 2 - Test bottle being wound on Lamtex Corp. machine. Note the Paraplast mandrel, partially wound, with windings being distributed from above, through the resin pot.

but it was found that the winding could be more accurately controlled on a hard surface, and that a rubber liner could be placed in a finished bottle after the mandrel was removed.

Fabrication of Test Bottles

The bottles were wound on a machine manufactured by the Lamtex Corp. (Fig. 2). This machine is capable of winding a bottle up to 18 in. in diameter and 6 ft in length. Phasing of the winding pattern is accomplished by changing a gear train, a chain drive, and the position of the resin pot. Extreme care was taken to phase in the machine for each series of windings. The glass is delivered to the winding machine from a creel which was modified to prevent abrasion of the glass and the accumulation of "fuzz balls" (agglomerations of abraded glass, which will disrupt the winding when passing through the resin pot, thereby causing defects if deposited on the mandrel). This modification was made by replacing all ceramic eyes with ball-bearing nylon rollers. The tension of the glass was controlled at 60 grams per end by hysteresis magnetic brakes and was frequently monitored with a Kidde-Sipp Tensometer. The resin temperature was controlled by a thermostat for both heating and cooling, as the case demanded. All windings were made in an air-conditioned room controlled at 73°F and 40 percent relative humidity.

The winding operation was closely observed. Any defects in the winding pattern, glass delivery, and resin application were noted. Bottles with known defects were not included in the test group. For this reason the data are selective, in that not every bottle that came off the winding machine was accepted for testing. After winding, the bottle was squeegeed to remove excess resin and placed on a rotating device, called a "B" stager, where it rotated at 73°F until the resin had gelled. A finished bottle is shown in Fig. 1.
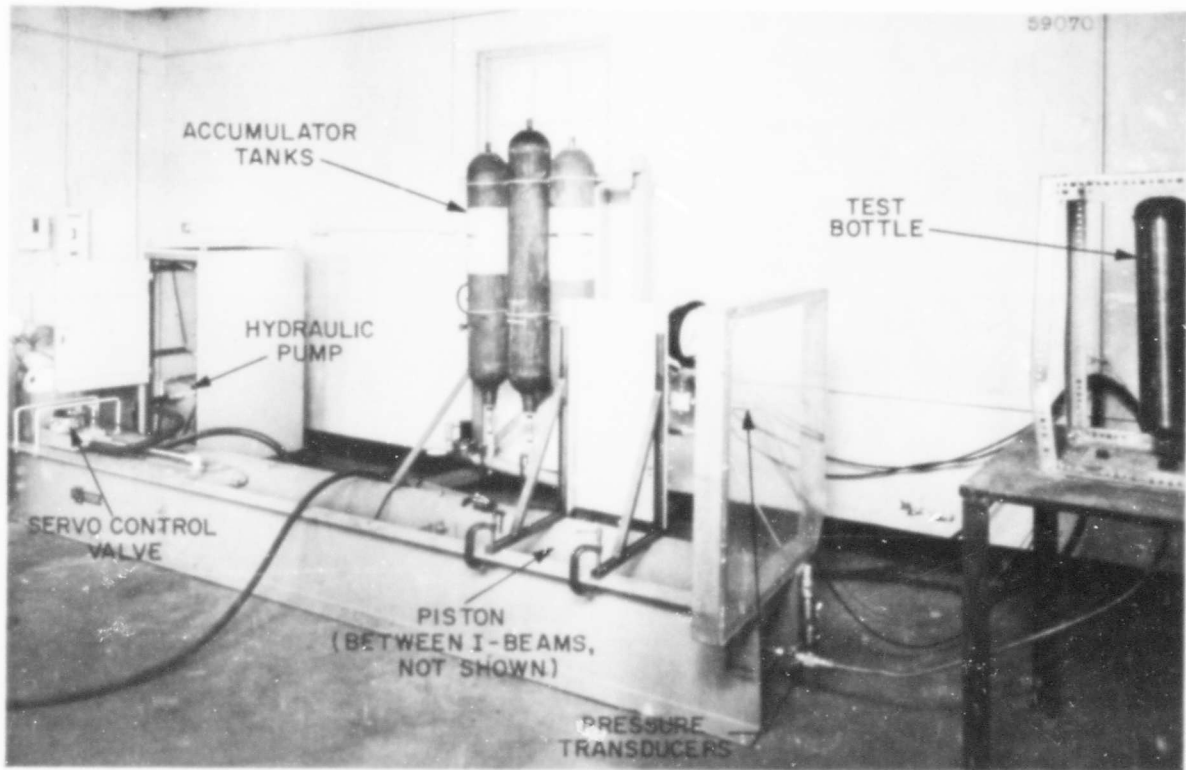
Fig. 3 - Bottle-test assembly

Curing the Bottles

All bottles that were to be cured at elevated temperatures were cured in an air-circulating oven while being rotated, so as to assure an even distribution of resin throughout the bottle when the resin softened during heating. The cure cycle and temperature for each resin were regulated by an automatic controller. Care was taken to baffle the oven so that a uniform temperature throughout the oven could be obtained. Upon completion of the cure schedule, the water-soluble Paraplast mandrels were washed from the bottles, which were then dried and stored at $73°$ F and 40 percent relative humidity.

Testing the Bottles

The bottles were tested on a hydraulically operated, servo-controlled machine which essentially consists of a hydraulic piston controlled by a command signal ramp function generator (Figs. 3,4). The pressure rate of increase was constant at 45 lb/in^2/sec for all tests. A liner was inserted into the bottle just prior to testing. The liner was a dipped rubber bladder, open at one end and in the same shape as the bottle, but smaller. The open end was taped to a fitting in such a manner that when the bottle was filled with water the bladder doubled back upon itself and thus sealed the taped connection. This simple liner proved to be very efficient and trouble free. A record of the testing was continuously made. Figure 5 shows characteristic bursts. The bottles were designed to be slightly unbalanced in the vertical windings, so as to force the burst in the cylindrical portion of the bottle, where a mathematical analysis of the burst stress can be more accurately applied.
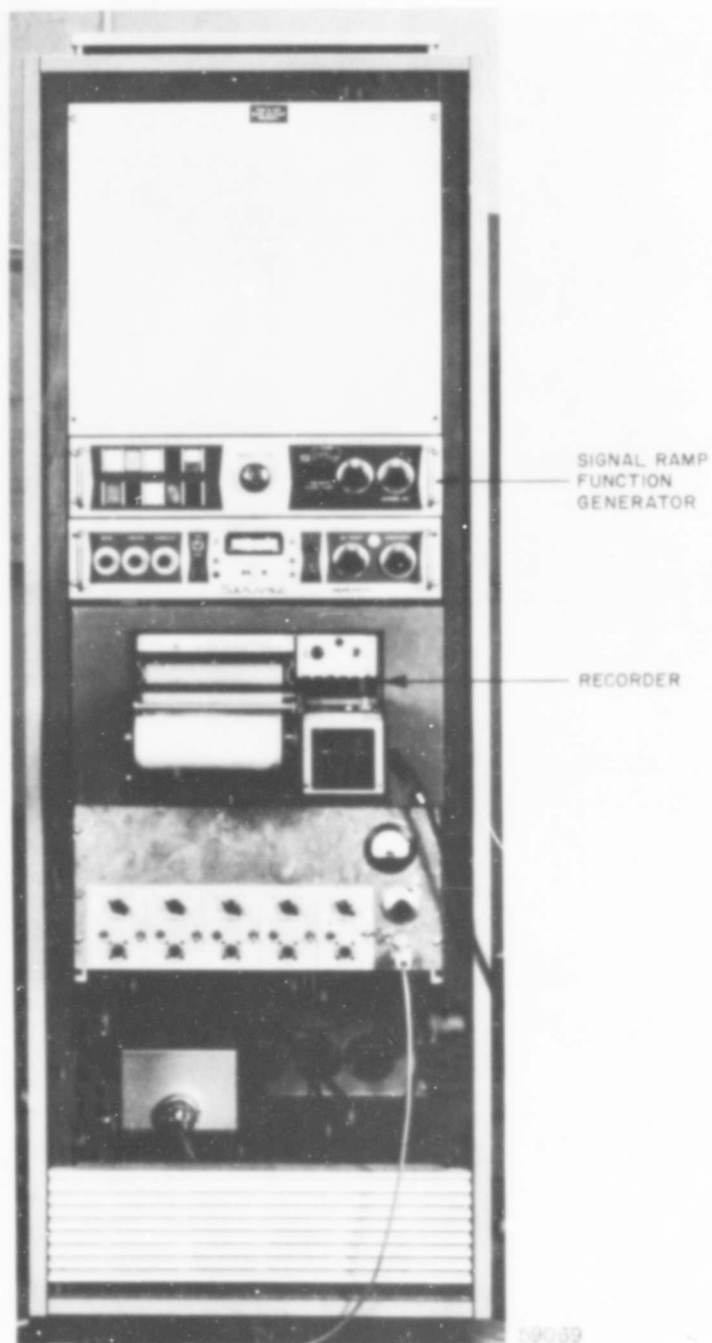
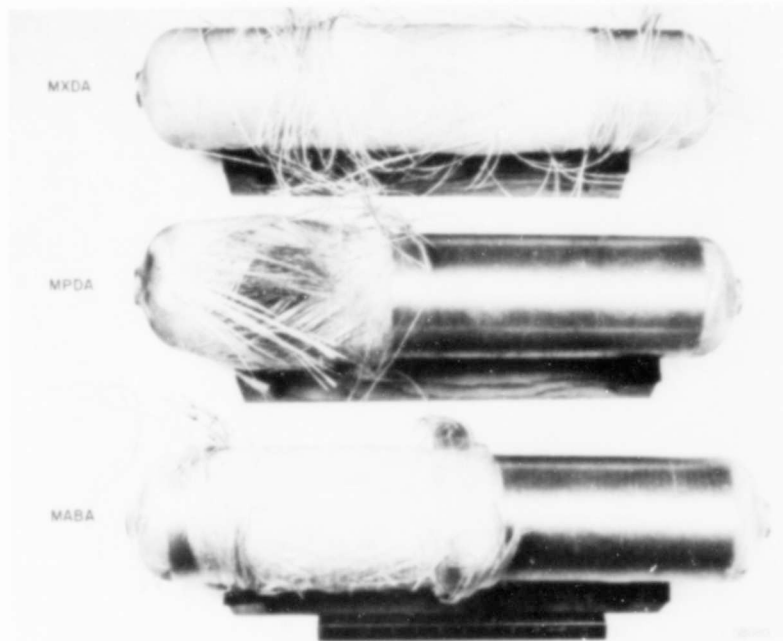Fig. 4 - Test-assembly control equipment

Fig. 5 - Characteristic bursts of bottles
wound with the three curing agents utilized
in this study

DISCUSSION OF THE DATA

There were six series of bottles wound and tested for this study. All of the bottles
were wound with the diglycidylether of bisphenol-A as the epoxy monomer. Two of the
groups of bottles, cured with MPDA, were wound for controls, i.e., to provide a base line
from which to evaluate the effects of the alterations of the curing agent and the resulting
tensile properties. Control 1 was wound at the onset of the project, and control 2 was
wound about three-fourths of the way through the project. The purpose of this second
control was to determine if a developing technique of bottle winding would place a sub-
stantial bias in the latter-wound bottles.

The data obtained from these six series of bursts are presented in Fig. 3. It can be
seen from these data that control 2 gave a higher burst level than did control 1. This
higher burst level means that as the manufacture of bottles proceeded, an improved
technique for winding a more perfect bottle was developed, which raised the burst level
slightly. This increase in the burst level amounts to about 4 percent. Since this bias
has been introduced, all the data must be evaluated with less certainty when only a few
percent separates comparisons.

Table 2 presents the mean burst pressures of the six groups of bottles and the tensile
properties of the resin used in each group. This table shows that the tensile properties
exhibited by the three cured resins differed considerably, while the mean burst level of
the bottles wound with these three resin systems varied but slightly. Since the variation
that was obtained between control 1 and control 2 is in the same percentage range as the
variations between the burst levels of bottles utilizing each of the three cured resin sys-
tems, it is concluded that the slight differences in burst levels are not meaningful.

Table 2
Tensile Properties of the Resin and Mean Burst Pressure of the Bottles

Curing Agent	Resin Tensile Properties				
	Mean Burst Pressure (psig)	Plastic Tensile (psi)	Ultimate Elongation (percent)	Elastic Modulus (psi)	Modulus of Toughness $\left(\dfrac{\text{in.-lb}}{\text{in.}^3}\right)$
MPDA					
Control #1	1780	12,800	7.5	463,000	610
Control #2	1860				
MXDA	1850	10,600	6.7	436,000	550
MABA	1900	12,200	10.5	409,000	1,000
MPDA (B-staged)	1700	Very low- too fragile to obtain data.			
MXDA (B-staged)	1730	Very low- too fragile to obtain data.			

When the significance of these data became apparent, it was decided that further tests were necessary to substantiate these results. Two additional groups of bottles were wound using MPDA and MXDA as the curing agent, but the bottles were "B" staged at room temperature only and were given no postcure. Epoxy resins which have been "B" staged at room temperature, using aromatic amines as the curing agent, are exceedingly weak. The "B"-staged plastics obtained with the use of MPDA and MXDA were so fragile that cast samples could not be removed from the mold without damage, and therefore it was not possible to determine their tensile properties. However, all of the tensile properties of "B"-staged resin are undoubtedly very low.

Table 2 shows that the "B"-staged MPDA bottles gave a mean burst pressure within 4 percent of the mean of the cured MPDA control 1 and within 8 percent of the cured control 2. Likewise, the "B"-staged MXDA bottles gave a mean burst pressure only 7 percent lower than the fully cured bottles. Figure 6, which presents the data spread for each group of bottles, shows that the highest single "B"-staged MPDA burst was above the highest single cured MPDA burst in control 1. Furthermore, the highest single burst of all the data was a "B"-staged MXDA bottle.
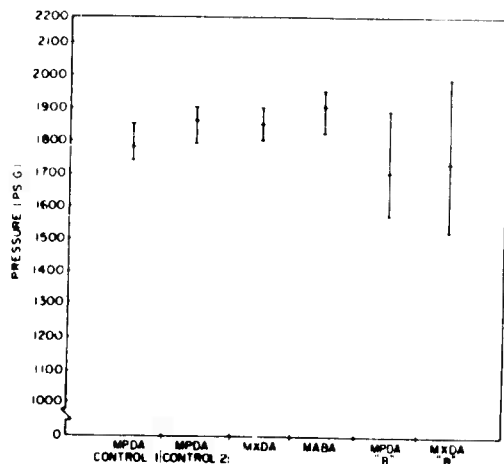
Fig. 6 - Burst data, high, mean, and low burst values are given for bottles would with curing agents used in the study

Figure 6 also shows that the data spread of burst pressures for "B"-staged bottles is substantially larger than the spread for cured bottles. The data spread is about 6 percent for the cured systems, while the data spread is 28 percent and 18 percent respectively for the "B"-staged MXDA and MPDA systems. All the data that were used to construct Fig. 6 are derived from bottles with no known defects either in winding or curing, or without variations in the bursting procedure. However, it is readily conceded that in any series of windings there will be defects that escape detection or are inherently undetectable. Therefore, a certain amount of data spread is expected. It is assumed, however, that the "B"-staged series will contain the same degree of imperfections as do the cured systems, since they were wound in an identical manner. It is apparent, from these facts and from the data as displayed in Fig. 6, that the "B"-staged resins have allowed these undetected imperfections to be more deleterious to the ultimate burst pressure than has been allowed by the cured systems.

The data from Table 2 and Fig. 6 indicate that a resin which is weak in all of its tensile properties does not greatly affect the mean burst level of a series of bottles. However, Fig. 6 demonstrates that a weak resin will allow defects in the bottle to be more deleterious and allow individual bottles to burst far below the design pressure. Also, the fact that some of the highest individual bursts were "B"-staged bottles indicates that the tensile properties of the resin are unimportant in a perfectly designed and fabricated bottle. These conclusions presuppose the use of a resin matrix capable of maintaining glass-pattern integrity and bottle geometry.

The only effect in a rocket-motor case of using a resin with poor tensile properties is to lower the mean burst level of a series of cases slightly. But more important in a rocket-motor case is the fact that the weak resin decreases the chance that any individual motor case will reach its designed burst pressure before failure. Hence, the use of a weak resin in the production of motor cases decreases the overall reliability of the cases. It is important, therefore, that a resin of the best properties be used in the production of motor cases in order to achieve greater reliability.

It is not known, at this time, what failure takes place in the "B"-staged resin but uncured that does not occur in the cured resin. However, the cured resin apparently has the ability to exert a healing effect on imperfections in the structure that the uncured resin does not have or has to a lesser degree. Of course, if the structure were perfectly designed and fabricated there would be no imperfections to be healed, and the resin would have no effect. The probability that commercially manufactured structures will be free of defects is very small, and therefore a suitably cured resin of good properties can be of benefit. The tests of the three cured systems showed no valid differences in their respective abilities to mitigate the effects of imperfections.

Calculations were made to determine the burst stress of these bottles in order to learn what appears to be a limiting value. A perplexing problem in calculating glass stress is the determination of the portion of load in the vertical direction that is borne by the helical windings. The bottles, having been designed to be unbalanced, always burst in the vertical windings. The test-bottle design used in this study had a 25-degree helix angle, from which the maximum stress in the vertical direction theoretically possible for the helicals to carry is slightly over 10 percent of the total glass stress. But it is believed, based on work at NRL and by others (2), that the helicals in practice carry somewhat less than the maximum possible load.

Therefore, the two extremes exist when the helicals carry no load and when they carry the maximum load theoretically possible in the vertical direction. As an arbitrary measure to simplify Table 3, the stress figures are expressed from calculations that assumed that the helicals carried 5 percent of the load in the vertical direction, or half the maximum load theoretically possible. Table 3 shows these stress levels for the mean burst strength of each of the six groups of bottles.

Table 3
Glass Stresses in the Burst Bottles

Resin Curing Agent	Burst Pressure (psig)			Glass Stress (psi)		
	Average	High	Low	Average	High	Low
MPDA, Control 1	1780	1850	1740	378,000	393,000	370,000
MPDA, Control 2	1860	1900	1790	395,000	404,000	380,000
MPDA, "B" Staged	1700	1890	1580	361,000	402,000	336,000
MXDA	1850	1900	1800	393,000	404,000	383,000
MXDA, "B" Staged	1730	1980	1520	368,000	420,000	324,000
MABA	1900	1950	1820	404,000	414,000	387,000

These calculated stress levels closely approach the measured tensile strength of "E" glass in strands and rovings. A figure of about 500,000 psi (3) is generally reported for the tensile strength of a single fiber, and about 400,000 psi (3) for strands of fibers. This figure of 400,000 psi for strands (or bundles of fibers) agrees well with the strength of a strand of fibers as predicted by the so-called "bundle theory" (3). Therefore, it is concluded that the bottles wound for this study show a high and nearly constant burst pressure because the quality of workmanship in their fabrication is such as to allow almost all of the glass to develop its maximum tensile stress before failure of the bottle. If this conclusion is correct, it means that little is to be gained in the way of improved ultimate burst pressures from further research to improve the resin properties. However, as previously indicated in this report, improvements in resin quality are a means for achieving greater reliability in reaching the burst level for which the structure is designed.

CONCLUSIONS

1. The tensile properties of the resin have an insignificant effect on the ultimate burst of an internally loaded, perfectly designed and fabricated filament wound vessel.

2. The properties of the resin do have an important role in reducing the variations in ultimate burst strengths of individual filament-wound vessels; hence, the resin is important in the reliability of the product.

3. Filament-wound vessels that have been carefully manufactured and inspected can develop glass stresses at burst that approach very nearly that of the theoretical tensile strength of the glass strands and rovings used in winding the vessel.

4. There is little prospect of increasing the average ultimate burst strength of filament-wound bottles by further research on the resin. This strength is largely controlled by the reinforcement. However, the reliability of the burst strength of pressure bottles, and rocket-motor cases, may be improved by additional resin research.

The above conclusions apply only to internally loaded vessels fabricated with commercial glass fibers and utilizing an epoxy matrix. These conclusions are further restricted to vessels which have not been previously stressed.

AN EVALUATION OF THE MATRIX AS IT AFFECTS
FILAMENT-WOUND REINFORCED PLASTICS

It is appropriate to summarize, at this time, some of the more important information that has been disclosed by research on filament-wound reinforced plastics, and to point out some of the remaining problems that must be solved in order to develop the full structural potential of these new materials.

First, this report establishes that glass-filament-reinforced composite structures may be wound which will develop, at burst, almost all of the tensile strength inherent in the glass. Other researchers have reported the ultimate tensile strength of "E" glass in bundles of fibers, as used in winding, to be about 400,000 psi (3). From the experiments described herein, as well as work elsewhere (4), it appears that any resin of good tensile properties will allow the glass in a wound structure to develop 90 percent or more of this ultimate tensile strength when loaded internally and when the fabrication is of best quality.

The process of fabricating a wound vessel does not appreciably damage the glass (3). However, imperfections resulting from strand breakage and agglomerations of abraded glass can and should be minimized by careful handling. The major problem in the fabrication of wound-glass-reinforced structures is that of quality control. The low burst pressures that are experienced with some vessels are probably attributable to a substantial variability in fabrication techniques rather than to inferior materials. Windings that have not been properly phased in, loss of tension on strands, and poor band formation are among the more common errors in winding.

The extent of damage to a motor case during proof testing must be determined. When a filament-wound vessel is loaded internally, as in a proof test, damage results. The nature and extent of this damage has not been determined. Research might find means of mitigating this damage or preventing it altogether.

The filament-wound structures that are produced today contain appreciable voids. The amount of voids reported by various organizations varies somewhat due to differences in impregnation procedures and the methods for determining the voids. Up to 3 to 4 percent voids by volume are probably innocuous in internally loaded vessels which are tested shortly after fabrication for ultimate burst strength, but such voids may well be deleterious to the structure that has weathered or otherwise been subjected to an atmosphere of high humidity. Voids appear to be an entirely different matter in structures that are subjected to an external load, i.e., compressive stresses (5). Data on externally loaded structures indicate that failure takes place in the resin through shear. Under these conditions the voids in the resin are a very important determinant of the performance of an externally loaded vessel. Research to eliminate voids in fiber-reinforced structures should be performed; it is essential to the maximum future development of these materials. The complete function of the resin in an externally loaded vessel has not been ascertained. This information is basic to any efficient design of a glass-reinforced plastic deep-submergence vessel. When the role of the resin is known, new resins could be developed to improve the capabilities of this application of glass-resin composites.

ACKNOWLEDGMENT

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13 ABSTRACT

A study has been conducted on amine-cured epoxy resins and the effects on the ultimate burst strength of internally loaded glass-reinforced filament-wound vessels when these resins were used as the plastic matrices. The tensile properties of the matrix were changed by systematically altering the molecular structure of the amine-cured epoxy resin. It was found that resin tensile properties had an insignificant effect on the ultimate burst strength of a well-designed and well-fabricated vessel. However, it was also found that a matrix of good tensile properties improved the reliability of the burst pressure by providing a healing effect on minor winding errors. The glass stresses at burst of the test vessels were in the 400,000-psi range, which is about the ultimate tensile strength of bundles of "E" glass.

A summary has been prepared which discusses the state of the art and unsolved problems relevant to the plastic matrix in glass-reinforced filament-wound vessels. In particular, the deleterious effect of voids on externally loaded structures and the necessity of removing these voids is discussed.

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Naval Research Laboratory. Report 6161.
FILAMENT-WINDING PLASTICS. PART 2 – ROLE OF THE RESIN IN GLASS-FIBER-REINFORCED STRUCTURES UNDER TENSILE STRESS, by F.S. Whisenhunt, Jr. 14 pp. and figs., December 1, 1964.

A study has been conducted on amine-cured epoxy resins and the effects on the ultimate burst strength of internally loaded glass-reinforced filament-wound vessels when these resins were used as the plastic matrices. The tensile properties of the matrix were changed by systematically altering the molecular structure of the amine-cured epoxy resin. It was found that resin tensile properties had an insignificant effect on the ultimate burst strength of a well-designed and well-fabricated vessel. However, it was also found that a

1. Filament wound construction – Materials – Tensile props.
2. Plastics – Tensile props.
I. Whisenhunt, F.S. Jr.

matrix of good tensile properties improved the reliability of the burst pressure by providing a healing effect on minor winding errors. The glass stresses at burst of the test vessels were in the 400,000-psi range, which is about the ultimate tensile strength of bundles of "E" glass.

A summary has been prepared which discusses the state of the art and unsolved problems relevant to the plastic matrix in glass-reinforced filament-wound vessels. In particular, the deleterious effect of voids on externally loaded structures and the necessity of removing these voids is discussed.

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